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U.S. DEPARTMENT OF COMMERCE
PATENT AND TRADEMARK OFFICE

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TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US)

09/889113

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/JP00/08040	15 November 2000 (15/11/00)	18 November 1999 (18/11/99)

TITLE OF INVENTION
POLYESTER YARN AND ITS METHOD OF PRODUCTION

APPLICANT(S) FOR DO/EO/US
Katsuhiko Mochizuki, Koji Sugano and Yuhei Maeda

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items under 35 U.S.C. 371:

1. ■ This express request to immediately begin national examination procedures (35 U.S.C. 371(f)).

2. ■ The U.S. National Fee (35 U.S.C. 371(c)(1)) and other fees as follows:

CLAIMS	(1) FOR	(2) NUMBER FILED	(3) NUMBER EXTRA	(4) RATE	(5) CALCULATIONS
	TOTAL CLAIMS	35 -20=	15	x $18.00	$ 270.00
	INDEPENDENT CLAIMS	2 -3=	0	x $80.00	
	MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ $270.00	270.00
	BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(4)): ☐ International preliminary examination fee paid to USPTO (37 CFR 1.482).....$690.00 ☐ No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)).....$710.00 ☐ Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO.....$1,000.00 ☐ International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2) to (4).....$ 100.00 ■ International Search Report enclosed $860.00				860.00
	Surcharge of $_____ for furnishing the National fee or oath or declaration later than ☐20 ☐30 mos. from the earliest claimed priority date (37 CFR 1.482(e)). $130.00				
			TOTAL OF ABOVE CALCULATIONS		1,400.00
	Reduction by ½ for filing by small entity, if applicable. Affidavits must be filed also. (Note 37 CFR 1.9, 1.27, 1.28.)				
			SUBTOTAL		1,400.00
	Processing fee of $_____ for furnishing the English Translation later than ☐20 ☐30 mos. from the earliest claimed priority date (37 CFR 1.482(f)). $130.00				
			TOTAL NATIONAL FEE		1,400.00
	Fee for recording the enclosed assignment (37 CFR 1.21(h)). $40.00				40.00
			TOTAL FEES ENCLOSED		$1,440.00

a. ■ A check in the amount of $1,440.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. 13-3405 in the amount of $_____ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ■ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 13-3405. A duplicate copy of this sheet is enclosed.

3. A copy of the International Application as filed (35 U.S.C. 371(c)(2)) JC18 Rec'd PCT/PTO 11 JUL 2001
   a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
   c. ■ has been transmitted by the International Bureau.

4. ■ A translation of the International Application into English (35 U.S.C. 371(c)(2)).

5. Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ have been transmitted by the International Bureau.

6. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).

7. ■ An oath or declaration of the inventor (35 U.S.C. 371(c)(4)).

8. ☐ A translation of the Annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Other document(s) or information included:

9. ☐ An Information Disclosure Statement under 37 C.F.R. 1.97 and 1.98.

10. ■ An Assignment document for recording and a Recordation Form Cover Sheet - Patents Only. Please mail the recorded assignment document to the person whose signature, name and address appears at the bottom of this page.

11. The above checked items are being transmitted
   a. ☐ before the 18th month publication.
   b. ■ after publication and the Article 20 communication but before 20 months from the priority date.
   c. ☐ after 20 months but before 22 months (surcharge and/or processing fee included).
   d. ☐ after 22 months (surcharge and/or processing fee included).
      Note: Petition to revive (37 C.F.R. 1.137(a) or (b)) is necessary if 35 U.S.C. 371 requirements submitted after 22 months *and no proper demand for International Preliminary Examination was made by 19 months from the earliest claimed priority date.*
   e. ☐ by 30 months and a proper demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
   f. ☐ after 30 months but before 32 months and a proper demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date (surcharge and/or processing fee included).
   g. ☐ after 32 months (surcharge and/or processing fee included).
      Note: Petition to revive (37 C.F.R. 1.137(a) or (b)) is necessary if 35 U.S.C. 371 requirements submitted after 32 months and a proper demand for International Preliminary Examination was made by 19 months from the earliest claimed priority date.

12. At the time of transmittal, the time limit for amending claims under Article 19
   a. ☐ has expired and no amendments were made.
   b. ☐ has not yet expired.

13. ☐ Certain requirements under 35 U.S.C. 371 were previously submitted by the applicant on _____, namely:

SCHNADER HARRISON SEGAL & LEWIS

Date: 11 July 2001 By: _____

T. Daniel Christenbury, Reg. No. 31,750
1600 Market Street, 36th Floor
Philadelphia, PA 19103

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Art Unit	:	**Customer No. 22469**
Examiner	:	
Serial No.	:	
Filed	: Herewith	Docket No.: 1232-01
PCT No.	: PCT/JP00/08040	
PCT Filed	: November 15, 2000	
Inventors	: Katsuhiko Mochizuki	
	: Koji Sugano	
	: Yuhei Maeda	
Title	: POLYESTER YARN AND	
	: ITS METHOD OF PRODUCTION	
		Dated: July 11, 2001

PRELIMINARY AMENDMENT

BOX PCT
Commissioner for Patents
Washington, DC 20231

Sir:

Prior to examination, using clean copies of the Specification and Claims and also marked-up versions of such, we respectfully request consideration of the following amendments and remarks:

In the Specification (Clean copy as amended)

Please replace the paragraph spanning pages 15 and 16 with the following:

Furthermore, the spinning oil applied will contain lubricant, emulsifier and antistatic agent, etc. Specifically, examples include mineral oils such as liquid paraffin, fatty acid esters such as octyl palmitate, lauryl oleate and isotridecyl stearate, dibasic acid diesters such as dioleyl adipate and dioctyl sebacate, esters of polyhydric alcohols such as trimethylolpropane trilaurate and coconut oil, aliphatic sulphur-containing esters such as lauryl thiodipropionate, nonionic surfactants such as polyoxyethylene oleyl ether, polyoxyethylene castor oil ether, polyoxyethylene nonyl phenyl ether and

trimethylolpropane trilaurate, anionic surfactants such as alkyl sulphonate and alkyl phosphate type metal salts or amine salts, sodium dioctylsulphosuccinate, sodium alkanesulphonate, etc, tetramethylene oxide/ethylene oxide copolymers, propylene oxide/ethylene oxide copolymers and the like, and there is employed a formulation which enhances the passage through the yarn production, warping and fabric production stages, in particular passage through the reeds and heddles at the time of weaving. Where required, there may also be used rust preventives, antibacterial agents, antioxidants, penetrating agents, surface tension lowering agents, phase reversal viscosity lowering agents, wear preventing agents and other such modifiers, or the like.

In the Claims (Clean copy as amended)

20. A method of producing polyester yarn according to Claim 15 which is characterized in that there is used a textured roll of surface roughness 1.5S-8S in the drawing and heat-treatment.

Remarks

We have amended the Specification and Claim 20 to make minor corrections to the PCT application that resulted from errors in translating the original Japanese text.

We respectfully request early examination on the merits.

Respectfully submitted,

T. Daniel Christenbury
Reg. No. 31,750
Attorney for Applicants

TDC:lh
(215) 563-1810

Version with Markings to Show Changes Made to the Specification

Please replace the paragraph spanning pages 15 and 16 with the following:

Furthermore, the spinning oil applied will contain lubricant, emulsifier and antistatic agent, etc. Specifically, examples include mineral oils such as liquid paraffin, fatty acid esters such as octyl palmitate, lauryl oleate and isotridecyl stearate, dibasic acid diesters such as dioleyl adipate and dioctyl sebacate, esters of polyhydric alcohols such as trimethylolpropane trilaurate and coconut oil, aliphatic sulphur-containing esters such as lauryl thiodipropionate, nonionic surfactants such as polyoxyethylene oleyl ether, polyoxyethylene castor oil ether, polyoxyethylene nonyl phenyl ether and trimethylolpropane trilaurate, anionic surfactants such as alkyl sulphonate and alkyl phosphate type metal salts or amine salts, sodium dioctylsulphosuccinate, sodium alkanesulphonate, etc, tetramethylene oxide/ethylene oxide copolymers, propylene oxide/ethylene oxide copolymers[, nonionic surfactants] and the like, and there is employed a formulation which enhances the passage through the yarn production, warping and fabric production stages, in particular passage through the reeds and heddles at the time of weaving. Where required, there may also be used rust preventives, antibacterial agents, antioxidants, penetrating agents, surface tension lowering agents, phase reversal viscosity lowering agents, wear preventing agents and other such modifiers, or the like.

Version with Markings to Show Changes Made to the Claims

20. A method of producing polyester yarn according to Claim [22] 15 which is characterized in that there is used a textured roll of surface roughness 1.5S-8S in the drawing and heat-treatment.

Specification

Polyester Yarn and its Method of Production

Technical Field

The present invention relates to polyester yarn comprising polytrimethylene terephthalate, and to its method of production. More particularly, it relates to polyester yarn and to a method of producing polyester yarn characterized in that yarn production can be carried out stably at high speeds without package tightening and with little variation in properties in the fibre lengthwise direction and, furthermore, when made into a fabric, there is little sense of tightness because it stretches at a low modulus, and it has a soft handle.

Background Art

Polytrimethylene terephthalate fibre is outstanding in its elastic recovery following elongation, possesses a low Young's modulus and soft bending characteristics and has good dyeing properties and, furthermore, chemically it has stable properties in the same way as polyethylene terephthalate. Hence, as may be seen for example from US Patent Nos 3,584,103 and 3,681,188, it has long been the subject of research as a potential clothing material.

However, the starting material 1,3-propanediol is comparatively expensive, so polytrimethylene terephthalate has not been used as a synthetic fibre hitherto.

In recent years, as disclosed for example in US Patent 5,304,691, a cheap method for the synthesis of 1,3-propanediol has been discovered, so the value of polytrimethylene terephthalate fibre has been re-examined.

According to investigations carried out by the present inventors, if the two-stage method generally employed in the case of polyethylene terephthalate fibre is applied as it is to polytrimethylene terephthalate, directly after spinning there commences a change in internal structure and, as a result of a phenomenon referred to as package tightening, differences in properties arise due to differences in the extent of such changes in internal structure between the package inner and outer layers, and so fibre of stable quality is not obtained.

As a means for resolving this problem, there has been proposed a method using DSD in which the spinning process and drawing process are conducted continuously and, prior to winding-up, the internal structure is subjected to heat setting, as described in JP-A-52-8123. However, even by this method it has not been possible to suppress package tightening completely.

Objective of the Invention

The present invention has as its objective to provide a polyester yarn which shows no package tightening in the yarn production process so that a package of stable product quality is obtained and, furthermore, which has a low Young's modulus in the elastic recovery region, and is outstanding in its soft stretch properties and softness; together with a method for the production of this polyester yarn.

Disclosure of the Invention

For the purposes of resolving the aforesaid problem, the polyester yarn of the present invention has the following constitution. Specifically, the present invention relates to

polyester yarn which is characterized in that it is a multifilament yarn substantially comprising polytrimethylene terephthalate, and as well as the strength from the stress-strain curve being at least 3 cN/dtex and the Young's modulus being no more than 25 cN/dtex, the minimum value of the differential Young's modulus at 3-10% extension is no more than 10 cN/dtex and the elastic recovery following 10% elongation is at least 90%.

Furthermore, this polyester yarn can be obtained by

a method of producing polyester yarn which is characterized in that multifilament yarn obtained by the melt spinning of polymer substantially comprising polytrimethylene terephthalate of intrinsic viscosity [η] at least 0.7 is hauled-off at a spinning rate of at least 2000 m/min and, without winding up, subjected to drawing and heat-treatment, after which it is continuously subjected to a relaxation heat treatment at a relaxation factor of 6 to 20% and wound-up as a package.

Moreover, woven fabric of the present invention has the following constitution. Specifically, it is

a woven fabric which is characterized in that the aforesaid polyester yarn is used as the warp yarn and/or the weft yarn

in the form of a twisted yarn of twist coefficient 10,000 to 20,000.

Brief Description of the Drawings

Figure 1: This is a schematic diagram showing an example of spin-drawing equipment for obtaining the polyester yarn of the present invention.

Figure 2: This is a schematic diagram showing another example of spin-drawing equipment for obtaining polyester yarn of the present invention.

Figure 3: This shows the stress-strain curve and the differential Young's modulus-strain curve for polyester yarn of the present invention (Example 1).

Figure 4: This shows the stress-strain curve and the differential Young's modulus-strain curve for polyester yarn lying outside the present invention (Comparative Example 4).

Explanation of the numerical codes

1: spinneret
2: cooling chimney
3: oiling guide
4: first heated roller
5: second heated roller
6: cooling roller
7: interlacing nozzle
8: winder

Best Mode for Carrying out the Invention

The polyester yarn of the present invention is multifilament yarn substantially comprising polytrimethylene terephthalate.

In the present invention, the polyester from which the polyester yarn is composed is polytrimethylene terephthalate (hereinafter abbreviated to PTT) where at least 90 mol% of the structural units are obtained from terephthalic acid as the chief acid component and 1,3-propanediol as the chief glycol component. However, there may be included copolymer components which can form other ester bonds, in a proportion which does not exceed 10 mol% and preferably does not exceed 6 mol%. Examples of copolymerizable compounds include dicarboxylic acids such as isophthalic acid, succinic acid, cyclohexanedicarboxylic acid, adipic acid, dimer acid, sebacic acid and 5-sodiumsulphoisophthalic acid, and diols such as ethylene glycol, diethylene glycol, dipropylene glycol, butanediol, neopentyl glycol, cyclohexanedimethanol, polyethylene glycol and polypropylene glycol, but there is to be no restriction to these. Moreover, optionally, there may be added titanium dioxide as a delustrant, fine silica or alumina particles as a lubricant, hindered phenol derivatives as an antioxidant, and colouring pigments, or the like.

It is important that the strength of the polyester yarn of the present invention be at least 3 cN/dtex. If the strength is less than 3 cN/dtex, as well as this leading to fuzzing and yarn breaks in subsequent processing stages such as weaving, the product obtained will also have reduced tear strength.

Furthermore, there is an inverse correlation between the extension at break and the frequency of occurrence of fuzzing at the time of weaving, and the higher the breaking extension

while still satisfying the requirement in terms of practical strength, the more the occurrence of fuzzing can be suppressed. Hence, the residual extension is preferably at least 40% and more preferably at least 45%.

Again, it is important that the polyester yarn of the present invention has a Young's modulus of no more than 25 cN/dtex and that it has a minimum value of differential Young's modulus at 3-10% extension of no more than 10 cN/dtex. These properties are closely related to the elongation characteristics and the elastic recovery characteristics in a stretch fabric, and in order to attain the soft stretch property which is the objective of the present invention it is preferred that these properties have low values. That is to say, by satisfying all the above properties, when in the form of a fabric there is easy initial stretch (low Young's modulus) and, furthermore, within the extension range of 3-10%, which is the practical stretch recovery region, elongation is possible with no resistance (low differential Young's modulus). Hence, it is possible to produce a soft stretch fabric which is outstanding in its comfort when worn.

The Young's modulus has a linear relationship to the flexural stiffness of the fabric, and the lower the Young's modulus the more outstandingly soft is the fabric handle. Hence, the Young's modulus is preferably no more than 22 cN/dtex and more preferably no more than 20 cN/dtex.

In the same way, the minimum value of the differential Young's modulus at 3-10% extension is preferably no more than 8 cN/dtex and more preferably no more than 5 cN/dtex.

The polyester yarn of the present invention has an elastic recovery of at least 90% following 10% elongation. If the elastic recovery is less than 90%, then there occurs the problem known as 'sagging' where, following elongation, there remains a portion which has undergone partial plastic deformation, so the woven material quality is reduced. The elastic recovery following 10% elongation is preferably at least 95% and more preferably at least 98%.

Now, the fact that yarn comprising PTT has outstanding elastic recovery is due to a considerable extent to its molecular structure. The reasons are thought to be because, in the crystal structure of PTT, the methylene chain of the alkylene glycol moiety has a gauche-gauche conformation, and interaction due to the stacking of benzene rings is low and the density low, so that flexibility is high, and hence the molecular chains readily stretch and recover by means of methylene chain rotation in the alkylene glycol moiety.

In experiments by the present inventors it was shown that the higher the degree of crystallinity the higher the elastic recovery. Consequently, the degree of crystallinity is preferably at least 30% and more preferably at least 35%. Here, the measurement of the degree of crystallinity was carried out based on the density in accordance with the density gradient column method of JIS L1013 (Chemical Fibre Filament Yarn Test Methods).

Furthermore, preferably, the boiling water shrinkage of the polyester yarn of the present invention is 3-15% and, moreover, the maximum value of the shrinkage stress is no more than 0.3 cN/dtex and the temperature at which the maximum value of shrinkage stress is shown is at least 120°C.

The boiling water shrinkage is one of the most important factors in terms of carrying out fabric design, and by making the boiling water shrinkage at least 3%, the setting properties are made favourable in subsequent processing stages, while by making it no more than 15% it is possible to obtain a fabric with a soft handle which is free of any sense of harshness. In the same way, if the heat shrink stress is too high, excess shrinkage will be introduced and the fabric handle will be harsh. Hence, in order to achieve a soft handle with no sense of harshness, the maximum value of the shrinkage stress is preferably no more than 0.3 cN/dtex and more preferably 0.15 to 0.25 cN/dtex. Again, the temperature at which the maximum value of shrinkage stress is shown is preferably at least 120°C and more preferably at least 130°C in order to facilitate subsequent processing such as setting and bulking-up.

In the case of the polyester yarn of the present invention, it is preferred that the CV% of the yarn lengthwise direction continuous shrinkage factor be no more than 5%. The CV% of the continuous shrinkage factor is an index of the uniformity of internal strain in the yarn lengthwise direction, and the smaller this value the higher the quality. In order to obtain fabric of high quality, the CV% is preferably no more than 5% and more preferably no more than 4%.

Again, it is preferred that the CF (coherence factor) value lies in the range 1-30, by subjecting the polyester yarn of the present invention to an interlacing treatment. Where the CF value is at least 1, it is possible to suppress single filament breaks at the time of yarn production and processing, and also at the time of weaving. Furthermore, where the CF

value is no more than 30, when for example performing combination to produce a combined yarn with different shrinkage as one component yarn, migration is facilitated, so this is preferred. It is further preferred that the CF value be 5 to 25.

The cross-sectional shape of the fibre from which the polyester yarn of the present invention is composed may be of circular cross-section, triangular cross-section, multilobal cross-section, flattened cross-section, X-shaped cross-section or other known profile section, and there are no particular restrictions thereon. Suitable selection may be made in accordance with the objectives.

Again, in order to enhance the softness when made into a woven fabric, the single filament fineness is preferably no more than 5 dtex and more preferably no more than 3 dtex.

In the case of the polyester yarn of the present invention, there is a strong correlation between the twist coefficient and the stretch property and, once the twist coefficient exceeds a fixed value, there is a tendency for the stretch property to rapidly increase. In practice, for a woven fabric employing yarn of twist coefficient about 5000, the percentage stretch is about 5%, but with a twist coefficient of 10,000 it is about 15% and with a twist coefficient of 14,000 it is about 30%. Hence, while the polyester yarn obtained in the present invention may be employed without twisting, it is more preferred that it be given a medium to hard twist with a twist coefficient of 10,000 to 20,000.

Now, the twist coefficient K is expressed by the relationship:-

twist coefficient $K = T \times D^{0.5}$

where, T = number of twists per metre of yarn length and D = yarn fineness (decitex).

Here, T, the number of twists per metre of yarn length, is the value determined by untwisting the yarn with an electrically-powered twist detector under a 90×10^{-3} cN/dtex load, and dividing the number of 'untwists' when the yarn is completely untwisted by the yarn length following untwisting.

The form of the fabric of the present invention may be that of a woven material, knitted material, nonwoven material or cushion material, etc, with suitable selection being made according to the objectives, and the fabric can be used in shirts, blouses, trousers, suits, blousons and the like.

Next, an example of the method of producing the polyester yarn of the present invention is provided.

As the method for producing the PTT which is the starting material for the polyester yarn of the present invention, there can be used a known method as it is. The intrinsic viscosity [η] of the PTT employed needs to be at least 0.7 in order to raise the spinnability at the time of yarn production and in order to obtain yarn of practical strength, but at least 0.8 is preferred.

Furthermore, in the production of the polyester yarn of the present invention, there may be employed continuous polymerization and spinning whereby, following the polymerization, the polymer is directly subjected to spinning

and drawing, or alternatively the polymer may first be converted into chip and dried, and then the spinning and drawing carried out.

The spinning temperature at the time of the melt spinning is preferably a temperature 10-60°C higher than the melting point of the PTT in order to stabilize the discharge from the spinneret, and more preferably the melt spinning is carried out at a temperature equal to the melting point plus 20 to 50°C. Again, in order to suppress oligomer deposition in the spinning and to enhance the spinning properties, there may be optionally provided 2-20 cm below the spinneret a heat shroud or suction device, or a means for generating an inert gas such as air, steam or nitrogen for preventing oxidative degradation of the polymer or spinneret contamination.

What is most important when producing the polyester yarn of the present invention is that there be employed the direct spin-draw method in which the drawing is immediately carried out following spinning, without temporarily winding-up.

In undrawn yarn comprising PTT, as stated above, a change in the internal structure begins right after spinning, with the phenomenon referred to as package tightening occurring, and this is a cause of differences in properties arising between the package inner and outer layers. When the present inventors carried out an investigation to suppress this package tightening, they found that an effective method comprises hauling-off the yarn at a spinning rate of at least 2,000 m/min and then, without temporarily winding up, immediately subjecting the yarn to drawing and heat treatment, after which it is continuously given a relaxation heat treatment by a relaxation factor of 5 to 20%. By using this

method, the problem of package tightening is markedly improved, and it is possible to obtain yarn of high quality in which differences between the package interior and exterior layers are extremely small. Moreover, it has also been discovered that, by subjecting the yarn to a relaxation heat treatment at a high relaxation factor, there is obtained soft stretch yarn which is easily stretched and has a low Young's modulus in the elongation recovery region.

In order to reduce yarn unevenness and obtain uniform yarn which does not tend to show defects such as dyeing variations, it is important that the spinning rate be at least 2,000 m/min. By raising the spinning rate, the spinning tension is raised, and by making the yarn less susceptible to the effects of external disturbances the draw-down behaviour is made stable. Hence, the spinning rate is preferably at least 3,000 m/min. Furthermore, in order to secure stable spinnability, it is preferred that the spinning rate be no more than 6,000 m/min.

Again, it is preferred that the draw ratio be set such that the residual extension is at least 40%.

It is important that the relaxation factor at the time of the relaxation heat treatment following the drawing be made at least 6 to 20% in order to obtain the polyester yarn which is the objective of the present invention. By carrying out a relaxation heat treatment of at least 6% following drawing, it is possible to accelerate the relaxation of internal strain in the fibre, so the level of delayed relaxation of the residual strain is low and package tightening is suppressed. Furthermore, as explained above, by the relaxation heat treatment, elongation is facilitated in the

practical extension range (up to 10% extension) and it is possible to confer outstanding characteristics in terms of soft stretch properties. It is further preferred that the relaxation factor be at least 8%. On the other hand, in order to achieve stability of yarn passage in the yarn production process, the relaxation factor is preferably no more than 20% and more preferably no more than 18%.

The relaxation heat treatment is now explained with reference to Figures 1 and 2.

Figure 1 is a schematic diagram of the method using a cooling roller in the relaxation heat treatment. Following discharge from spinneret **1**, cooling is carried out in chimney **2**, then convergence and oiling effected at oiling guide **3** and the yarn hauled-off and the temperature raised by first heated roller **4**, after which drawing and heat setting are performed between first heated roller **4** and second heated roller **5**. Furthermore, after passing through the drawing process, by employing the heat of second heated roller **5**, a relaxation heat treatment is carried out between the second heated roller **5** and cooling roller **6**, and winding-up performed by winder **8**. Now, in order to conduct the relaxation heat treatment still more efficiently, carrying out the relaxation treatment using a heat treatment means employing hot air or steam as a heating medium between the second heated roller **5** and cooling roller **6**, or carrying out the relaxation treatment in two stages by providing a third heated roller, are effective means for realizing the objective of the present invention.

Figure 2 is a schematic diagram of a method employing an interlacing nozzle in the relaxation heat treatment, and

interlacing nozzle 7 has the role of a yarn cooling device and of a tension gradient controller. That is to say, by means of the interlacing treatment it is possible to lower the yarn tension prior to interlacing, so by utilizing the shrinkage stress produced by the heat of second heated roller 5 it is possible to perform a relaxation heat treatment between the second heated roller 5 and interlacing nozzle 7. In such circumstances, the relaxation factor can be controlled by varying the actuating air pressure of the interlacing nozzle. Again, the relaxation treatment may also be carried out using a heat treatment means employing hot air or steam as a heating medium between the second heated roller 5 and interlacing nozzle 7, or in two stages by providing a third heated roller.

In each case the relaxation factor is readily controlled and they are methods which are favourably employed in obtaining the polyester yarn of the present invention.

In the case of the heated roller (the second heated roller in the examples illustrated in Figure 1 and Figure 2) which serves both for the drawing and heat setting and for the relaxation heat treatment, it is preferred that there be used a textured roller of surface roughness 1.5S to 8S. The surface roughness is the section value of the maximum height (R_{max}) described in JIS B0601, and 1.5S to 8S in practice corresponds to the section values 1.6S, 3.2S, 6.3S. In terms of maximum height, this corresponds to more than 0.8 μm and up to 6.3 μm. By making the surface roughness at least 1.5S, the frictional coefficient between the yarn and roller is considerably reduced and there is a suitable degree of slip, so even at a high relaxation factor there is no winding of the yarn back on the heating roller, and stable yarn

production is possible. As the surface roughness becomes higher, so yarn passage becomes more stable in the relaxation process but, if it exceeds 8S, the yarn surface is excessively abraded so a reduction in strength is brought about. The surface roughness of the heated roller is more preferably 3.2S to 6.3S (R_{max}: 1.7-6.3 μm). Now, the surface roughness is determined from measurement of the maximum height R_{max} using a Hommel Tester model T1000, made by the Hommel Co., based on JIS B0601.

In order to produce yarn stably without yarn breaks, the drawing temperature (the temperature of the first heated roller) is preferably 10-50°C higher than the glass transition temperature of the PTT, and more preferably the drawing is carried out at the glass transition temperature plus 20 to 40°C. The heat setting and relaxation heat treatment temperature (the temperature of the second heated roller) should be set within the range 90-180°C so as to achieve the desired percentage heat shrinkage but, in order to effect uniform relaxation of the residual stresses formed by the drawing, a temperature in the range 105-180°C is more preferred.

Furthermore, the spinning oil applied will contain lubricant, emulsifier and antistatic agent, etc. Specifically, examples include mineral oils such as liquid paraffin, fatty acid esters such as octyl palmitate, lauryl oleate and isotridecyl stearate, dibasic acid diesters such as dioleyl adipate and dioctyl sebacate, esters of polyhydric alcohols such as trimethylolpropane trilaurate and coconut oil, aliphatic sulphur-containing esters such as lauryl thiodipropionate, nonionic surfactants such as polyoxyethylene oleyl ether,

polyoxyethylene castor oil ether, polyoxyethylene nonyl phenyl ether and trimethylolpropane trilaurate, anionic surfactants such as alkyl sulphonate and alkyl phosphate type metal salts or amine salts, sodium dioctylsulphosuccinate, sodium alkanesulphonate, etc, tetramethylene oxide/ethylene oxide copolymers, propylene oxide/ethylene oxide copolymers, nonionic surfactants and the like, and there is employed a formulation which enhances the passage through the yarn production, warping and fabric production stages, in particular passage through the reeds and heddles at the time of weaving. Where required, there may also be used rust preventives, antibacterial agents, antioxidants, penetrating agents, surface tension lowering agents, phase reversal viscosity lowering agents, wear preventing agents and other such modifiers, or the like.

From the point of view of passage through subsequent processing stages, the amount of oil applied is preferably 0.3 to 1.2 wt% in terms of the yarn.

Examples

Below, the present invention is explained in further detail by means of examples. Now, the various property values in the examples were determined by the following methods.

A. Intrinsic Viscosity [η]

Using an Ostwald viscometer, sample polymer was dissolved in o-chlorophenol (abbreviated below to OCP) and the relative viscosity η_r determined at a number of points, after which the value at infinite dilution was obtained by extrapolation.

B. Strength, Extension and Young's Modulus (initial resistance to stretching)

The sample was subjected to measurement using a Tensilon UCT-100 produced by the Orientec Co., under constant rate of elongation conditions as described in JIS L1013 (Chemical Fibre Filament Yarn Test Methods). The breaking extension was determined from the elongation at the point showing the maximum tenacity in the S-S curve.

Furthermore, the Young's modulus was measured under the conditions given for the initial resistance to stretching in 7.10 of JIS L1013 (Chemical Fibre Filament Yarn Test Methods).

C. Differential Young's Modulus

This was determined by differentiation of stress with respect to extension at points on the S-S curve obtained in B.

D. Elastic Recovery

Using a Tensilon UCT-100 produced by the Orientec Co. and with the clamp spacing at 20 cm, the sample was stretched to 10% of the clamp spacing at a rate of extension of 10 cm/min, then the load immediately removed at the same rate, and the elastic recovery determined from the hysteresis curve recorded.

elastic recovery (%) = $(\beta/\alpha) \times 100$

α: elongation when stretched 10%

β: recovered elongation up to the point when the stress equals the initial load

E. Shrinkage Stress

Measurement was carried out at a rate of temperature rise of 2.4°C/sec using a thermal stress measurement device produced by Kanebo Engineering (Co.). The sample comprised a 2 x 10 cm loop and the initial tension = fineness (decitex) x 0.9 x (1/30) gf.

F. CV% of Continuous Shrinkage Factor in Yarn Lengthwise Direction

Using an FTA500 made by the Toray Engineering Co., measurement was carried out with the set tension = fineness (decitex) x 0.9 x (1/60) gf, treatment temperature = 100°C (under steam), yarn velocity = 10 m/min and sample length = 10 m. The shrinkage was recorded on a chart and the CV% of the continuous shrinkage factor in the yarn lengthwise direction determined.

G. CF Value

Measurement was carried out under the conditions shown for the Degree of Interlacing in 7.13 of JIS L1013 (Chemical Fibre Filament Yarn Test Methods). The CF value (coherence factor) was determined from the average value L (mm) of the length of the interlace using the following formula, based on 50 measurements.

$$\text{CF value} = 1000/L$$

H. Degree of Crystallinity

The density was measured in accordance with the Density Gradient Column Method in 7.14.2 of JIS L1013 (Chemical Fibre

Filament Yarn Test Methods) and the degree of crystallinity obtained by the following formula.

$$X_c\ [\%] = \{d_c \times (d - d_a)\}/(d \times (d_c - d_a)\} \times 100$$

X_c: degree of crystallinity (%)

d: measured yarn density

d_c: density of completely crystalline region

d_a: density of completely amorphous region

Here, d_c = 1.387 g/cm^3, d_a = 1.295 g/cm^3

Example 1

Using the spin-draw machine shown in Figure 1, homo-PTT of intrinsic viscosity [η] 0.96 was melted and spun from 24-hole spinneret **1** at a spinning temperature of 265°C and, after cooling in chimney **2** and then converging and oiling at oiling guide **3**, haul-off was performed at 3,000 m/min by means of first heated roller **4**, and having raised the temperature of the yarn by five laps at 70°C, drawing was carried out by means of second heated roller **5** at a drawing rate of 4800 m/min (draw ratio = 1.6). After heat-setting by five laps at 140°C, relaxation was performed by a relaxation factor of 10% between second heated roller **5** and cold roller **6**, and then while performing an interlacing treatment at an actuating pressure of 0.2 MPa using interlacing device **7**, wind-up was performed at 4220 m/min with winder **8**, and 54 decitex, 24 filament, drawn yarn obtained. Now, for second heated roller **5** there was used a textured roll of surface hardness 3.2S (R_{max}: 3 μm).

The yarn production characteristics were good and there were no yarn breaks or filament wrap-around. Furthermore, the strength of the polyester yarn obtained was 3.6 cN/dtex, the Young's modulus (initial resistance to stretching) was 20.8 cN/dtex, the minimum value of the differential Young's modulus at an extension of 3-10% was 1.8 cN/dtex, and the elastic recovery following 10% elongation was 97.8%. The physical properties are shown in Table 1, and the stress-strain curve and the differential Young's modulus-strain curve are shown in Figure 3.

Moreover, when weaving was carried out as a 1/4 twill using this multifilament yarn as the warp/weft, the weaving characteristics and the woven material quality were good and the material possessed light stretchability.

Example 2, Example 3

The same conditions were employed as in Example 1 except that the drawing rate was either 4350 m/min (draw ratio = 1.45) [Example 2] or 5000 m/min (draw ratio = 1.67) [Example 3]. The polyester yarn of Example 2 had a strength of 3.3 cN/dtex, which was lower than that of Example 1. Other characteristics were good in the same way as in Example 1. Moreover, while in the case of the polyester yarn of Example 3 the number of machine stoppages at the time of weaving increased to about twice when compared to Example 1, other properties were good.

Example 4, Example 5

The same conditions were used as in Example 1 except that the relaxation factor between the second heated roller 5 and the

cold roller **6** was made 6% [Example 4] or 18% [Example 5]. The polyester yarns of Example 4 and Example 5 were good in terms of their yarn production properties and woven material quality in the same way as in Example 1, and they had light soft stretchability. In particular, the woven material of Example 5 was even more outstanding in its softness than that of Example 1.

Comparative Example 1

The same conditions were used as in Example 1 except that there was employed homo-PTT of intrinsic viscosity [η] 0.68. The spinnability of the polyester of Comparative Example 1 was poor and there were numerous yarn breaks in the drawing zone, so sampling was impossible.

Comparative Example 2

The same conditions were used as in Example 1 except that the drawing rate was made 3900 m/min (draw ratio = 1.3). The polyester yarn of Comparative Example 2 had low strength and high extension, the strength being 2.9 cN/dtex and the extension 73.5%, and furthermore its elastic recovery following 10% stretching was low and the practical durability after forming a fabric was poor.

Comparative Example 3, Comparative Example 4

The same conditions were used as in Example 1 except that the relaxation factor between the second heated roller **5** and the cold roller **6** was made 22% or 3%. In the case of the polyester yarn of Comparative Example 3 where the relaxation factor was 22%, there was considerable yarn oscillation over

the second heated roller and, furthermore, yarn breaks occurred with yarn twisting around the second heated roller.

In the case of Comparative Example 4 where the relaxation factor was 3%, differences in properties arose between the package inner layer and outer layer due to occurrence of package tightening, and there were variations in thickness matching the package end face period. Furthermore, the weaving properties were poor and the quality of the dyed product was bad. Again, while the fabric possessed stretchability, it exhibited elongation characteristics where stretching was extremely difficult. The physical properties are shown in Table 1, and the stress-strain curve and the differential Young's modulus-strain curve are shown in Figure 4.

Comparative Example 5

The same conditions were used as in Example 1 except that the drawing rate was made 5250 m/min (draw ratio = 1.75), cold roller **6** was removed and the relaxation factor was made 0%. In the case of Comparative Example 5, there was marked package tightening exceeding even that of Comparative Example 4 and, furthermore, the fabric obtained had stretch characteristics in which elongation was extremely difficult, and it was inferior too in its softness.

Example 6

The same conditions were used as in Example 1 except that the first heated roller **4** velocity was made 1000 m/min and the second heated roller **5** velocity was made 3500 m/min (draw ratio = 3.5). Fabric comprising the polyester yarn of

Example 6 showed good stretch characteristics in the same way as in Example 1, but dyeing unevenness arose in the dyed fabric which was thought to be due to yarn unevenness.

Example 7

The same conditions were used as in Example 1 except that the second heated roller **5** was changed to a 0.8S (R_{max}: no more than 0.8μm) mirror surface roll. In the case of Example 7, the travelling yarn in the relaxation zone between the second heated roller and cold roller **6** was unstable, and oscillation occurred on the second heated roller, with winding back on the roller and numerous yarn breaks occurring. Hence, compared to Example 1 the number of yarn breaks was about 10-fold.

Example 8

The polyester yarn obtained in Example 1 was subjected to 2000 t/m (twist coefficient K: 14700) S/Z twisting to produce warp and weft yarns, and then a 1/4 twill fabric was produced. This was subjected to relaxation scouring at 98°C by the usual method, and then intermediate setting carried out at 160°C. Subsequently, 15 wt% weight reduction was carried out with hot aqueous 3% NaOH solution, dyeing then performed and finish setting carried out. The fabric obtained was soft and its stretch properties were extremely outstanding.

In the table, 'relaxation factor' refers to the 'relaxation factor between the second heated roller and the cold roller **6**'; the 'differential Young's modulus' refers to the 'minimum value of differential Young's modulus at an extension of 3 to

10%'; the 'elastic recovery' refers to the 'elastic recovery following 10% elongation'; the 'shrinkage stress' refers to the 'maximum value of shrinkage stress'; the 'peak temperature' refers to the 'temperature showing the maximum value of shrinkage stress'; the 'shrinkage CV%' refers to the 'CV% of the lengthwise direction continuous shrinkage'; and the 'woven fabric quality' refers to the 'quality of the appearance of the woven fabric after dyeing (functional evaluation)'.

Industrial Applicability

With regard to the polyester yarn of the present invention and its method of production, as well as there being no package tightening in the yarn production stage and the package having a stable quality, it is possible to obtain woven fabric of low Young's modulus in the elastic recovery region and which is outstanding in its soft-stretch properties and softness.

Table 1

		Examples					Comparative Examples					Examples	
		1	2	3	4	5	1	2	3	4	5	6	7
Intrinsic viscosity	[η]	0.96	0.96	0.96	0.96	0.96	0.68	0.96	0.68	0.96	0.96	0.96	0.96
First HR velocity	m/min	3000	3000	3000	3000	3000	3000	3000	3000	3000	3000	1000	3000
Second HR velocity	m/min	4800	4350	5000	4800	4800	4800	3900	4800	4800	5250	3500	4800
Relaxation factor	%	10.0	10.0	10.0	6.0	18.0	10.0	10.0	22.0	3.0	0.0	10.0	10.0
Strength	cN/dtex	3.6	3.3	3.7	3.7	3.4	-	2.9	-	3.5	3.8	3.7	3.6
Extension	%	50.5	59.2	43.2	42.0	57.8	-	73.5	-	44.3	26.5	45.4	50.1
Young's modulus	cN/dtex	20.8	19.4	21.5	21.7	19.8	-	18.9	-	21.5	28.6	21.0	20.8
Differential Young's modulus	cN/dtex	1.8	1.5	2.5	6.6	1.4	-	1.4	-	11.2	14.4	3.2	2.0
Elastic recovery	%	97.8	90.8	98.0	98.2	93.3	-	85.5	-	98.5	98.8	98.1	97.2
Degree of crystallinity	%	38	36	39	40	37	-	36	-	43	47	40	38
Boiling water shrinkage	%	6.7	6.2	7.5	8.0	6.5	-	5.8	-	8.7	10.0	7.3	6.6
Shrinkage stress	cN/dtex	0.17	0.13	0.19	0.20	0.15	-	0.11	-	0.25	0.33	0.19	0.17
Peak temperature	°C	168	169	170	170	167	-	167	-	171	171	172	167
Shrinkage CV%	%	2.8	3.7	3.0	3.8	3.2	-	4.2	-	7.6	7.9	5.2	4.5
CF value		9.5	14.5	8.2	4.7	16.9	-	15.4	-	0.8	0.2	4.1	13.8
Woven fabric quality	4-stage evaluation	◎	O	◎	O	◎	-	O	-	X	X	Δ	Δ
Stretch properties	4-stage evaluation	◎	◎	◎	O	O	-	Δ	-	X	X	◎	◎

Claims

1. Polyester yarn which is characterized in that it is a multifilament yarn substantially comprising polytrimethylene terephthalate, and as well as the strength from the stress-strain curve being at least 3 cN/dtex and the Young's modulus being no more than 25 cN/dtex, the minimum value of the differential Young's modulus at 3-10% extension is no more than 10 cN/dtex and the elastic recovery following 10% elongation is at least 90%.

2. Polyester yarn according to Claim 1 which is characterized in that the Young's modulus is no more than 22 cN/dtex.

3. Polyester yarn according to Claim 1 which is characterized in that the minimum value of the differential Young's modulus at 3-10% extension is no more than 5 cN/dtex.

4. Polyester yarn according to Claim 1 which is characterized in that the residual extension is at least 45%.

5. Polyester yarn according to Claim 1 which is characterized in that the elastic recovery following 10% elongation is at least 95%.

6. Polyester yarn according to Claim 1 which is characterized in that the degree of crystallinity is at least 30%.

7. Polyester yarn according to Claim 1 which is characterized in that the boiling water shrinkage is 3-15% and, furthermore, the maximum value of the shrinkage stress is no more than 0.3 cN/dtex and the temperature at which the maximum value of shrinkage stress is shown is at least 120°C.

8. Polyester yarn according to Claim 7 which is characterized in that the maximum value of the shrinkage stress is 0.15 to 0.25 cN/dtex.

9. Polyester yarn according to Claim 7 which is characterized in that the temperature at which the maximum value of shrinkage stress is shown is at least 130°C.

10. Polyester yarn according to Claim 1 which is characterized in that the CV value of the continuous shrinkage in the yarn lengthwise direction is no more than 5%.

11. Polyester yarn according to Claim 1 which is characterized in that the CF value is 1-30.

12. Polyester yarn according to Claim 11 which is characterized in that the CF value is 5-25.

13. Polyester yarn according to Claim 1 where the fineness of the individual filaments from which the polyester yarn is composed is no more than 3 dtex.

14. A woven fabric which is characterized in that polyester yarn according to any of Claims 1 to 13 is

used as the warp yarn and/or the weft yarn in the form of a twisted yarn of twist coefficient 10,000 to 20,000.

15. A method of producing polyester yarn which is characterized in that multifilament yarn obtained by the melt spinning of polymer substantially comprising polytrimethylene terephthalate of intrinsic viscosity [η] at least 0.7 is hauled-off at a spinning rate of at least 2000 m/min and, without winding up, subjected to drawing and heat-treatment, after which it is continuously subjected to a relaxation heat treatment at a relaxation factor of 6 to 20% and wound up as a package.

16. A method of producing polyester yarn according to Claim 15 which is characterized in that there is carried out the melt spinning of polytrimethylene terephthalate of intrinsic viscosity [η] at least 0.8.

17. A method of producing polyester yarn according to Claim 15 which is characterized in that the spinning is carried out at a temperature 20-50°C higher than the melting point of the polytrimethylene terephthalate.

18. A method of producing polyester yarn according to Claim 15 which is characterized in that it is hauled-off at a spinning rate of at least 3,000 m/min.

19. A method of producing polyester yarn according to Claim 15 which is characterized in that the relaxation heat treatment is carried out at a relaxation factor of 8 to 18%.

20. A method of producing polyester yarn according to Claim 22 which is characterized in that there is used a textured roll of surface roughness 1.5S-8S in the drawing and heat-treatment.

21. A method of producing polyester yarn according to Claim 15 which is characterized in that there is used a textured roll of surface roughness 3.2S-6.3S in the drawing and heat-treatment.

22. A method of producing polyester yarn according to Claim 15 which is characterized in that the drawing temperature is 10-50°C higher than the glass transition temperature of polytrimethylene terephthalate.

23. A method of producing polyester yarn according to Claim 15 which is characterized in that the heat setting and relaxation heat treatment are carried out at a temperature in the range 105-180°C.

Abstract

The present invention relates to polyester yarn which is characterized in that it is a multifilament yarn substantially comprising polytrimethylene terephthalate, and as well as the strength from the stress-strain curve being at least 3 cN/dtex and the Young's modulus being no more than 25 cN/dtex, the minimum value of the differential Young's modulus at 3-10% extension is no more than 10 cN/dtex and the elastic recovery following 10% elongation is at least 90%.

Furthermore, said polyester yarn can be obtained by a method of producing polyester yarn which is characterized in that multifilament yarn obtained by the melt spinning of polymer substantially comprising polytrimethylene terephthalate of intrinsic viscosity [η] at least 0.7 is hauled-off at a spinning rate of at least 2000 m/min and, without winding up, subjected to drawing and heat-treatment, after which it is continuously subjected to a relaxation heat treatment at a relaxation factor of 6 to 20% and wound up as a package.

Moreover, the present invention also relates to a woven material of outstanding soft-stretchability which is characterized in that the aforesaid polyester yarn is used as the warp yarn and/or the weft yarn in the form of twisted yarn of twist coefficient 10,000 to 20,000.

In this way, it is possible to produce yarn stably at a high yarn production rate without package tightening occurring, and, as well as there being little variation in properties in the fibre lengthwise direction, when

made into fabric, said fabric stretches at low modulus so there is little sense of tightness, and it is possible to provide polyester yarn and woven materials with a soft handle.

Figure 1

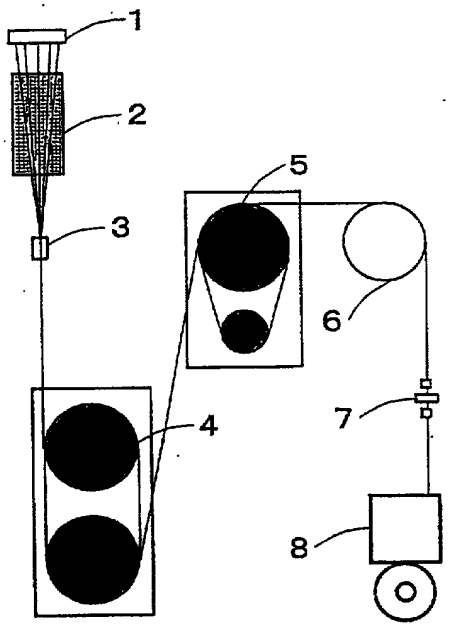


Figure 2

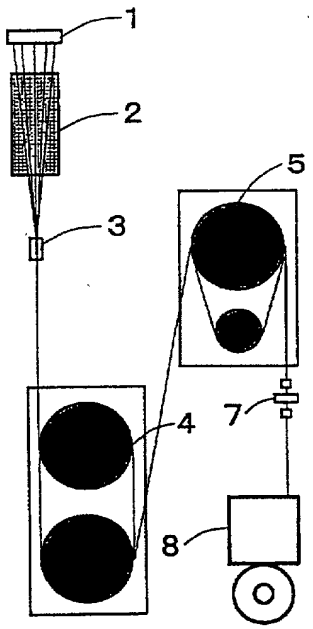
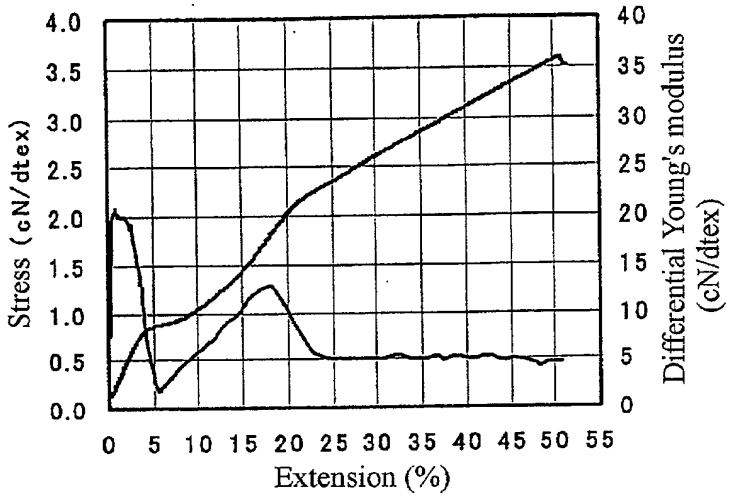
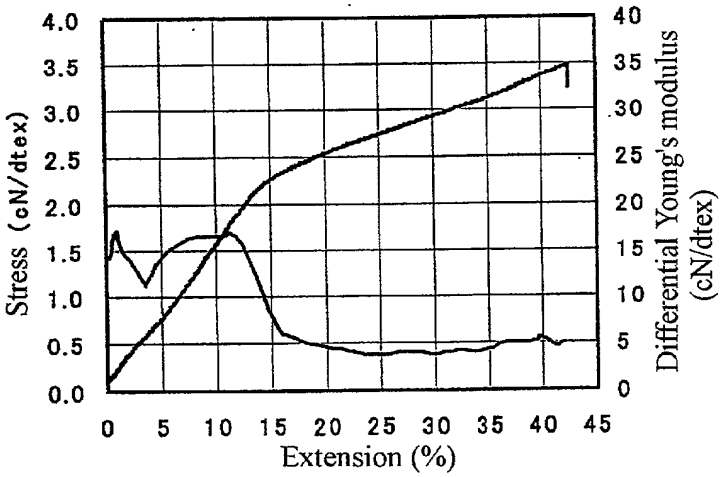


Figure 3



Polyester yarn of this invention (Example 1)

Figure 4



Polyester yarn outside scope of this invention
(Comparative Example 4)

Attorney Docket No. 1232-01

- ☐ Original Application
- ☑ PCT National Application
  U.S. Designated Office
- ☐ Continuation or Divisional Application
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As a below named inventor, I hereby declare that:

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I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled POLYESTER YARN AND ITS METHOD OF PRODUCTION

☒ which is described in the specification and claims

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Application Serial No. _____

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I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

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Number	Country	Date of Filing (day,month,year)	Priority Claimed
327943/99	Japan	18 November, 1999	■ yes ☐ no
			☐ yes ☐ no
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			☐ yes ☐ no
			☐ yes ☐ no

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(Application Serial No.)	*(Filing Date)*	*(Status)(patented,pending,abandoned)*

POWER OF ATTORNEY: As a named inventor, I hereby appoint the registered attorneys listed under Customer No. 022469 and the following registered attorneys to prosecute this application and transact all business in the United States Patent and Trademark Office connected therewith:

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(Page 3)

Attorney Docket No. 1232-01

I hereby petition for grant of a United States Letters Patent on this invention.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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